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# HPLC ANALYSIS OF HELICOPTER ROTOR BLADE MATERIALS

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POLYMER RESEARCH BRANCH

March 1990

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER MTL TR 90-12	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle)  HPLC ANALYSIS OF HELICOPTER ROTOR BLADE MATERIALS		5. TYPE OF REPORT & PERIOD COVERED  Final Report
7. AUTHOR(s)  Gary L. Hagnauer and David A. Dunn		6. PERFORMING ORG. REPORT NUMBER
9. PERFORMING ORGANIZATION NAME AND ADDRESS  U.S. Army Materials Technology Laboratory Watertown, Massachusetts 02172-0001 SLCMT-EMP		8. CONTRACT OR GRANT NUMBER(s)
11. CONTROLLING OFFICE NAME AND ADDRESS  U.S. Army Laboratory Command 2800 Powder Mill Road Adelphi, Maryland 20783-1115		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS  D/A Project: 1L162105.AH84
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12. REPORT DATE March 1990
		13. NUMBER OF PAGES 37
		15. SECURITY CLASS. (of this report)  Unclassified
16. DISTRIBUTION STATEMENT (of this Report)  Approved for public release; distribution unlimited.		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number)		
Liquid chromatograph , Quality assurance Adhesives Helicopter blades Chemical analysis Epoxies Testing Resins Rubber		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number)  (SEE REVERSE SIDE)		

Block No. 20

ABSTRACT

This report describes high performance liquid chromatography (HPLC) test methods developed for the quality assurance of organic materials used in the manufacture of Army helicopter rotor blades. Since the chemical compositions of most of the organic materials were unknown at the start of this project, HPLC was employed to help identify components in specimens. Test methods were developed and then applied to monitor the chemical compositions of specimens received from the manufacturer during blade fabrication. Six HPLC test methods were used to evaluate eight sets of specimens of 12 different materials received over a span of three years. The HPLC test methods included size-exclusion, normal-phase, and reverse bonded-phase chromatography techniques and employed a variety of columns, mobile phases, and detectors. Major changes in chemical composition and variations in relative concentrations of chemical components were noted in seven of the 12 materials monitored during the course of this study. Test results are summarized and recommendations are made.

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## INTRODUCTION

This report describes high performance liquid chromatography (HPLC) test methods developed for the quality assurance of organic materials used in the manufacture of certain helicopter rotor blades. The test methods were designed to monitor the chemical compositions of commercial materials procured and used in blade production.

HPLC is one of the most versatile and economically viable approaches for analyzing soluble organic materials. Sample solutions are injected into a liquid mobile phase, which is pumped through column(s) packed with a stationary phase to facilitate separation, and then into a detector. The detector monitors the concentrations of the separated components, and its signal response recorded as a function of time after sample injection provides a **finger print** of the sample's chemical composition. Quantitative information may be obtained if sample components are known, and if standards for the components are available. Recent advances have resulted in improved and automated HPLC instrumentation that is relatively low cost and simple to operate and maintain.

The chemical compositions of most of the organic material specimens were unknown at the start of this project. Eight sets of specimens of 12 different materials were received for analysis over a span of three years. The specimens were stored at -15°C and only removed for sampling. Descriptions of the specimens are given below and the specimen numbers are given in Table 1.

**Epon 828** - A clear or pale yellow liquid epoxy resin manufactured by Shell Chemical Company and having diglycidyl ether of bisphenol A isomers and oligomers as primary components.

**BDMA/MTHPA** - A dark amber, low viscosity liquid which is a mixture of methyl tetra hydrophthalic anhydride (MTHPA) with benzyl dimethylamine (BDMA) in a ratio of 80:1 w/w MTHPA:BDMA.

**Spline Winding Resin** - An aromatic epoxy resin designated XB2793.

**Spline Curing Agent** - An aromatic amine manufactured designated XU205.

**Skin Material** - An epoxy resin S-2 glass prepreg.

**STA 66 Doubler** - An epoxy resin prepreg consisting of a hybrid tape (T300 graphite/S-2 glass).

**Adhesive A** - An epoxy resin film adhesive used for blade bonding.

**Primer A** - A primer used with film Adhesive A. The specimen is a liquid and has a yellow precipitate.

**Adhesive B** - An epoxy resin film adhesive used for spar bonding.

**Primer B** - A primer used with film Adhesive B. The specimen is a liquid and has a yellow precipitate.

**Abrasion Boot** - Uncured polyurethane sheet.

**Balance Weight Adhesive** - Uncured neoprene sheet.

Table 1. HELICOPTER BLADE SPECIMEN NUMBERS

Sample Set #	1	2	3	4	5	6	7	8
Date Received	0 Month	4 Months	7 Months	14 Months	22 Months	29 Months	31 Months	36 Months
Epon 828	1AC1	1AF2	1AF3	1AF4	1AC5	1AF6	1AF7	-
BDMA/MTHPA	BM1	BM2	BM3	BM4	BM5	BM6	-	-
Spline Resin	2AA1	-	-	2AC2	-	2AC3	-	-
Spline Curing Agent	2AB1	-	2AD2	2AD3	2AB4	2AD5	-	-
Skin Material	3AA1	3AB2	3AB3-4	3AB5	3AA6	3AB7	3AB8	3AB9
STA 66 Doubler	4AA1	4AB2	4AB3	4AB4-5	4AA6	4AB7	4AB8	4AB9
Adhesive A	5AG2-3	5AG9	5AG10	5AG11	5AE12	5AG13	5AG14	5AG15
Primer A	5AG1P	5AG2P	5AG3P	5AG4P	5AG5P	-	5AG6P	-
Adhesive B	5AF1	5AH2	5AH3	5AH4	5AH5	-	-	-
Primer B	5AH1P	5AH2P	5AH3P	5AH4P	5AH5P	-	-	-
Abrasion Boot	-	5AB1-3	-	5AB4-7	-	5AB8	5AB9	-
Balance Weight	5AC1,5AD2	-	5AD3	5AD4	5AC5	5AD6	-	5AD7

### EXPERIMENTAL

A Waters Associates ALC/GPC-244 instrument with M6000A solvent delivery systems, M720 system controller, 710B WISP auto-injection system, R-400 differential refractive index (RI) detector, M440 dual-UV detector, and M730 data module was used for the HPLC analyses. Reagent grade water was prepared from distilled water using a Millipore Milli-Q2 water purification system. Distilled-in-glass iso-octane (C8) and UV grade tetrahydrofuran (THF) was used as received from Burdick & Jackson Laboratories.

Liquid specimens were warmed to room temperature and mixed before sampling. Thin sections were cut from the elastomeric specimens to obtain samples. To obtain representative prepreg and film adhesive samples, 2 x 6 inch sections were cut from each specimen, weighed, and extracted with THF. The remaining prepreg fibers and film adhesive backing material were then dried and weighed to determine the weight-percent (w%) fiber and concentrations of the prepreg resin and film adhesive solutions. Solutions were prepared in volumetric flasks with THF as the solvent and filtered through 0.2 micrometer Millipore membrane filters. The nominal concentrations of the solutions were 1.0% w/v or 10 micrograms/microliter.

The following HPLC test conditions were developed for quality assurance of the blade materials.

Procedure:	SEC-1
Technique:	size exclusion chromatography (SEC)
Columns:	4 micro-Styragel (1000, 500, 2 x 100 Å) or 5 micro-Styragel (1000, 2 x 500, 2 x 100 Å)
Concentration:	10 micrograms/microliter
Inject Volume:	20 microliters
Flow Rate:	2 mL/min
Mobile Phase:	THF
Detectors:	RI, or UV214 nm, or UV254 nm, or UV280 nm
Analysis Time:	25 minutes

Procedure: SEC-2  
Technique: size exclusion chromatography  
Columns: 4 micro-Styragel ( $10^6$ ,  $10^5$ ,  $10^4$ ,  $10^3$  Å) or  
2 Zorbax PSM-1000-S and PSM-60-S  
Concentration: 0.2 micrograms/microliter  
Inject Volume: 100 microliters  
Flow Rate: 2 mL/min or 1.5 mL/min  
Mobile Phase: THF  
Detectors: RI or UV254 nm  
Analysis Time: 25 minutes  
Calibration: ASTM Standard Method D3516 or D3593

Procedure: HPLC-1  
Technique: normal phase HPLC  
Columns: 2 micro-Porasil  
Concentration: 10 micrograms/microliter  
Inject Volume: 20 microliters  
Flow Rate: 2 mL/min  
Mobile Phase: (60% C8/40% THF) to 100% THF  
30 min, gradient 6  
Detectors: UV254 nm or UV280 nm  
Analysis Time: 35 minutes

Procedure: RPHPLC-1  
Technique: reverse bonded-phase HPLC  
Column: micro-BondapakC18  
Concentration: 10 micrograms/microliter  
Inject Volume: 15 microliters  
Flow Rate: 2 mL/min  
Mobile Phase: (60% H<sub>2</sub>O/40% THF) to 100% THF  
30 min, gradient 6 or 25 min, gradient 6  
or  
(55% H<sub>2</sub>O/45% THF) to (15% H<sub>2</sub>O/85% THF)  
30 min, gradient 5  
Detector: UV280 nm  
Analysis Time: 35 minutes

Procedure: RPHPLC-2  
Technique: reverse bonded-phase HPLC  
Column: micro-BondapakC18 Radial Pak with  
Waters RCM-100 Radial Compression Module  
Concentration: 10 micrograms/microliter  
Inject Volume: 20 microliters  
Flow Rate: 2 mL/min  
Mobile Phase: (60% H<sub>2</sub>O/40% THF) to 100% THF  
25 min, gradient 6  
Detector: UV280 nm  
Analysis Time: 30 minutes

Procedure: RPHPLC-3  
 Technique: Dicyandiamide (Dicy) Analysis Method  
 Column: micro-BondapakC18  
 Concentration: 10 micrograms/microliter  
 Inject Volume: 20 microliter  
 Flow Rate: 2 mL/min  
 Mobile Phase: H<sub>2</sub>O  
 Detector: UV230 nm (Perkin-Elmer LC75 variable UV detector)  
 Analysis Time: 2 minutes

In the initial phase of the program, the first three sets of specimens were analyzed using test procedures SEC-1, SEC-2, HPLC-1, and RPHPLC-2. The test procedures were then modified to improve resolution and reduce analysis times, and only selected procedures were applied to analyze particular samples. The recommended test procedures are indicated in Table 2.

Table 2. RECOMMENDED TEST PROCEDURES

Sample	SEC-1	SEC-2	HPLC-1	RPHPLC-1	RPHPLC-2	RPHPLC-3
Epon 828	X			X	X	
BDMA/MTHPA	X			X		
Spline Resin	X			X		
Spline Curing Agent				X		
Skin Material	X			X		X
STA 66 Doubler	X		X	X		
Adhesive A	X		X	X		
Primer A	X			X		
Adhesive B	X			X		
Primer B	X			X		
Abrasion Boot	X	X				
Balance Weight	X	X				

## RESULTS AND DISCUSSION

In general, the organic material portions of the specimens were fully soluble in THF. The HPLC chromatograms shown in Figures 1, 2, and 3 were obtained from the analysis of a film Adhesive A sample. The HPLC chromatograms are recorder traces of detector signal versus time. The positions (retention times) and the heights, or areas, of the peaks provide a fingerprint of chemical composition. For a given set of experimental conditions, each component of the resin has a characteristic retention time and detector response. The retention time is a function of the separation mechanism, and the size of the peak is proportional to the amount of the component in the sample. Differences in composition may be revealed when chromatograms are overlaid. The absence of peaks, appearance of new peaks, and changes in peak size indicate disparities in composition. A variety of techniques and detectors may be employed. For a particular material, certain procedures may be preferred for fingerprinting while special methods may need to be developed for analyzing specific components.

Size exclusion chromatography involves the separation of molecular species according to their size in solution. The separation takes place predominantly in the pores of the column packing where the molecules permeate into and out of pores to a greater or lesser extent depending upon their sizes and upon the distribution of pore sizes available to them. The larger the size of a molecule in solution, the shorter its retention time will be since the total pore volume available to it will be smaller. Hence, for the conditions used in Figure 1, components having a molecular weight (MW) greater than about 500 g/mol will have retention times between 10 and 14 minutes. Components having the same size in solution will have the same retention time, but may have quite different detector responses.

The fingerprints in Figure 2 illustrate the application of normal phase HPLC. In this case, the mobile phase is usually a solvent of low or intermediate polarity, and separation depends upon specific interactions between the solute molecules and the surface of a polar-packing material, e.g., silica. Retention times increase with the polarity of the solute molecules and are highly dependent upon selection of the mobile phase. If the mobile phase is not sufficiently polar, extremely long retention times, peak tailing, and irreversible absorption on the column packing may occur. If the mobile phase is too polar, poor resolution is obtained. To improve resolution, solvent programming techniques may be used to adjust the polarity of the mobile phase during analysis. The chromatograms in Figure 2 were obtained by programming the mobile phase along a linear gradient over a period of 30 minutes. Again, it is noted that the use of several detectors provide more detailed fingerprints and clues to molecular differences in components. For example, significant differences in the molecular structures of components producing peaks at 7.1, 9.1, and 9.5 minutes in Figure 2 are expected because of the great disparity between their UV absorbance characteristics at 254 and 280 nm.

Reverse bonded-phase HPLC is a type of liquid partition chromatography in which a relatively nonpolar stationary phase is chemically bonded to silica-support material and the mobile phase is polar. Separation is based upon the relative solubility and distribution of the solute between the mobile and bonded phases, such that solutes which are more soluble in the bonded phase, or poorly soluble in the mobile phase, tend to have longer retention times. The main advantage of this technique is that the column packing is quite stable and may accommodate a variety of mobile phases and operating conditions. Excellent resolution is achieved, especially when solvent programming is employed, as shown in Figure 3.

The fingerprints in Figures 1, 2, and 3 are complementary, i.e., if one procedure does not detect a difference in composition, another might. Indeed, if certain components are not monitored because of poor detector response, or insolubility, differences in their concentrations may indirectly be observed by changes in the sizes of the peaks for components that are detectable. The epoxy resin monomer p,p'-diglycidyl ether of bisphenol A (DGEBA) was identified as a component in film Adhesive A by its retention time and detector response. The presence of DGEBA was verified by running a standard.

Results from the HPLC quality assurance of organic materials used in the manufacture of the helicopter rotor blade are summarized below.

#### **Epon 828**

Epon 828 contains at least 30 components resolvable by HPLC. The p,p' and o,p isomers of the DGEBA monomer ( $n = 0$ ), the DGEBA oligomers ( $n = 1$  and  $2$ ), and a

dihydroxy DGEBA hydrolysis product are identified as major components and may be quantitatively analyzed. There are only very slight differences (approximately 1%) in the compositions of the seven Epon 828 specimens. It is unlikely that such differences have a significant affect on the processability or performance of composites manufactured with the resin. The recommended test procedures are SEC-1 and RPHPLC-1 (Figures 4 and 5).

### **BDMA/MTHPA**

The chemical compositions of the BDMA/MTHPA mixtures are appreciably different from one another. There are at least 10 components resolvable by HPLC, and reaction products between MTHPA and BDMA are apparent. SEC with RI detection is an excellent technique for monitoring the concentration of MTHPA. Comparing the compositions of BM1 and BM2, SEC analysis (Figure 6) shows that 67% MTHPA in sample BM1 has reacted, perhaps via hydrolysis, compared to 20% in sample BM2. Such a large difference in MTHPA content would have an affect on epoxy resin polymerization reactions which use the BDMA/MTHPA curing agent and might have a significant affect, not only on resin processability, but also on the properties and performance of the cured resin.

Reverse bonded-phase HPLC is also a good technique for the quality assurance of BDMA/MTHPA. BDMA may be quantitatively analyzed using RPHPLC, and reproducible chromatograms are obtained. Differences in BDMA/MTHPA samples are obvious when their RPHPLC fingerprints are compared (Figure 7). The use of a UV214 nm detector is recommended for the analysis of MTHPA.

Test procedures SEC-1 and RPHPLC-1 are recommended for the quality assurance of BDMA/MTHPA samples. It is also recommended that the test procedures be applied as soon as possible after the BDMA/MTHPA mixtures are prepared, and before they are used, since MTHPA is susceptible to hydrolysis. The composition of the mixtures will change with time and will depend upon storage and handling conditions.

### **Spline Winding Resin**

At least 15 HPLC resolvable components are present in the spline winding resin. HPLC analysis indicates that the resin contains a DGEBA epoxy resin, and that the DGEBA monomer concentration is approximately 60 w%. Differences in the compositions of the spline winding resin samples were detected using HPLC and were attributed to poor sampling techniques. The spline winding resin tends to form a precipitate, perhaps due to crystallization of some of the components. Unless the resin is heated and well mixed before sampling and solution preparation, HPLC analysis will show compositional differences. The same components are present in all the samples; however, the percentages of each component may vary widely due to the precipitation effect. This effect is illustrated in Figure 8. The DGEBA monomer is indicated in the RPHPLC chromatogram of the resin (Figure 9). Test procedures SEC-1 and RPHPLC-1 are recommended for the spline winding resin.

### **Spline Curing Agent**

The spline curing agent has at least 15 HPLC resolvable components. Upon comparison of chromatograms (Figure 10) differences in relative peak heights indicate that there are appreciable specimen-to-specimen variations in the concentrations of curing agent components. However, it cannot be stated without more detailed structure-property information whether

the differences affect resin processability or properties of the cured resin. Procedure RPHPLC-1 is recommended for quality assurance.

### Skin Material

There are over 30 HPLC resolvable components in the resin portion of the skin material. HPLC analysis shows that the initial skin material specimens contained a DGEBA resin, the curing agent dicyandiamide, and a high molecular weight polymeric material ( $2100 < MW < 32,000$  g/mol) among other components. Procedure SEC-3 was applied to estimate the molecular weight range of the high molecular material, and procedure RPHPLC-4 is recommended for the quantitative analysis of dicyandiamide. The dicyandiamide content was found to be 3.40% w/w in the resin portion of the skin material. The glass fiber content ranged from 62% to 66% w/w.

The chemical compositions of the first six specimens were essentially identical with a variance in composition of only  $\pm 2\%$  for major components. However, HPLC analysis showed that the composition of specimen 3AB7 was quite different from those of the first six specimens, and that specimens 3AB8 and 3AB9 had compositions similar to 3AB7. Reverse bonded-phase HPLC clearly illustrates the extent to which the compositions are different (Figure 11). Differences are also apparent from the SEC analysis shown in Figure 12. Upon comparison, the results suggest that specimens 3AB7 have added low molecular weight components, and that an intermediate resin component in 3AA1 through 3AA6 has been replaced with a somewhat higher molecular weight epoxy resin in 3AB7 through 3AB9. Also, it is noted that the average resin content in specimens 3AA1 through 3AA6 was 34.4 w%; whereas the resin contents of 3AB7, 3AB8, and 3AB9 were 37.7, 39.7, and 36.0 w%, respectively. It is concluded from the HPLC analysis that either the supplier altered the formulation of the skin material resin or the rotor blade manufacturer changed suppliers after the 22-month specimen.

Test procedures SEC-1, RPHPLC-1, and RPHPLC-3 are recommended for quality assurance of the skin material resin.

### STA 66 Doubler

There are more than 30 components in the STA 66 doubler specimen resolvable by HPLC (Figures 13a and 13b). The specimens consist of unidirectional glass and graphite fibers impregnated with resin. The overall resin content is approximately 38 w%. According to SEC analysis (Procedure SEC-2), a high molecular weight component is present in the resin portion and has MW values ranging from 2400 to 120,000 g/mol.

The chemical compositions of the first six specimens, 4AA1 through 4AA6, were similar to one another, but quite different from those of the later specimens. The differences are clearly evident when RPHPLC and SEC chromatograms of specimens 4AA1, 4AA6, and 4AB7 are compared. From the appearance of the chromatograms shown in Figure 14, it is suspected that there was a major change in the resin formulation after specimen 4AA6 (22 months). It is also noted that variations in the relative heights of the two peaks indicated in the RPHPLC chromatograms of 4AA6 and 4AB7 (see Figure 14) suggest that either the prepreg supplier has modified the resin composition or the rotor blade manufacturer changed suppliers after the 22-month specimen.

SEC analysis, as shown in Figures 15 and 16, supports the RPHPLC results even though a different separation mechanism is involved. The resin contents of specimens 4AB4, 4AA6, and 4AB7 were 34.4, 37.7, and 39.7 w%, respectively. Procedures SEC-1, HPLC-1, and RPHPLC-1 are recommended for quality assurance.

#### Film Adhesive A

There are more than 30 components in film Adhesive A which are resolvable by HPLC. Test procedures for the adhesive were discussed earlier in this section, as shown in Figures 1, 2, and 3, and were applied for quality assurance of the film adhesive specimens. Since there was no change in the general pattern of the HPLC fingerprints, i.e., peak retention times and detector responses were similar for all specimens, it is concluded that there were no changes in the types of components used in the adhesive formulation. However, when HPLC chromatograms are examined closely, variations in the relative amounts of components become apparent. For example, the reverse bonded-phase HPLC chromatograms (Procedure RPHPLC-1) of five specimens are compared in Figure 17. Although the five specimens have similar fingerprints, differences in the relative sizes of peaks located at 12.2, 13.2, 14.7, 15.8, and 16.8 minutes are evident.

Variations in composition may be determined quantitatively if components are identified and standards are available for calibration. Using purified DGEBA monomer (the peak retention time for the monomer is 7.2 minutes) as a standard, the weight percentages of DGEBA in the resin of the film adhesive specimens were evaluated from the integrated DGEBA peak areas. For most specimens, the average concentration of DGEBA ( $n = 0$ ) was found to be within the range of 20 to 24 w%. However, some specimens, as shown in Table 3, were found to have DGEBA concentrations well outside the normal range.

Table 3. RPHPLC ANALYSIS OF FILM ADHESIVE A SPECIMENS

Specimen	W% DGEBA	Area Ratios		
		A1/A0	A2/A0	A3/A0
5AG2 (0 Month)	20.3	0.974	0.552	0.287
5AG10 (7 Months)	21.8	0.928	0.713	0.344
5AE12 (22 Months)	17.5	0.816	0.805	0.404
5AG13 (29 Months)	21.8	0.941	0.575	0.314
5AG14 (31 Months)	19.6	0.914	0.672	0.342
Average	20.2	0.915	0.663	0.338
STD DEV	1.6	0.053	0.093	0.037
% STD DEV	7.9%	5.8%	14.0%	10.9%

If the components are unknown, or if standards are unavailable for calibration, integrated peak area ratios may be compared to estimate specimen-to-specimen reproducibility. For example, the integrated peak areas for components with peak retention times of 7.2 (A1), 11.7 (A2), and 13.9 (A3) minutes are divided by the integrated area of the DGEBA peak (A0) to obtain the peak area ratios shown in Table 3. From this treatment of data, it is estimated that the specimen-to-specimen percent standard deviation (% STD DEV) in the relative concentration of major components is approximately 10%. Since the % STD DEV

for the repetitive HPLC analysis of a single specimen is less than 1%, reverse bonded-phase HPLC is an excellent technique for monitoring compositional differences.

Additional information on the possible causes and effects of the compositional variations may be gained by comparing SEC chromatograms. Since SEC chromatograms of the adhesive specimens appear to be nearly identical (Figures 18a and 18b) the chromatograms must be overlaid (Figure 18c) to discern differences. As shown in Figure 18, the relative amount of low MW resin component(s) (the peak retention time is 21.74 minutes) is somewhat less in 5AG12 than in 5AG2, and the portion of the chromatogram representing high MW resin components extends to a higher MW for 5AG12 than for 5AG2. This observation suggests that specimen 5AG10 is further advanced than 5AG2 and, consequently, that 5AG10 probably has a higher viscosity and may process differently than 5AG2.

More work relating chemical composition to processability and ultimate properties of the cured adhesive is required to determine the significance of the variations in adhesive composition and to develop criteria for quality assurance. Procedures RPHPLC-1 and SEC-1 are recommended for quality assurance. The normal phase procedure HPLC-1 may also be applied if precautions are taken to properly reequilibrate the HPLC column after each analysis.

#### **Primer A**

Over 20 HPLC resolvable components are present in Primer A. No changes in components were detected and variations in the relative amounts of components were small over the three-year period. SEC and RPHPLC chromatograms are shown in Figures 19 and 20. Procedures SEC-1 and RPHPLC-1 are recommended for quality assurance.

#### **Film Adhesive B**

Over 20 HPLC resolvable components are present in film Adhesive B. During the two-year period that Adhesive B specimens were monitored, no changes in adhesive components were detected and variations in the concentrations of the major components were similar to those observed in film Adhesive A. Comparison of SEC and RPHPLC chromatograms of Adhesive B (Figures 21 and 22) with those obtained for Adhesive A, as shown in Figures 1 through 3, show that the two film adhesives have quite different chemical compositions. Procedures SEC-1 and RPHPLC-1 are recommended for quality assurance.

#### **Primer B**

There are more than 20 HPLC resolvable components in Primer B. The primer contains DGEBA and a high MW component with molecular weights ranging from 3000 to 18,000 g/mol as estimated by SEC (Procedure SEC-1) analysis. There is also an insoluble residue in the primer which introduces problems in sample preparation, i.e., THF solutions of the primer tend to clog membrane filters even after they are centrifuged. The filtration behavior suggests that microgel is present in the primer and makes representative sampling difficult for HPLC analysis.

No changes in primer components were detected during the two-year period that primer specimens were monitored. Slight differences in the relative amounts of primer components were noted, but it was not possible to tell whether the differences were real or consequences of the sampling problem.

SEC and RPHPLC chromatograms (Figures 23 and 24) of Primer B are very different from those of Primer A (see Figures 19 and 20) indicating that the two primers have different compositions. Procedures SEC-1 and RPHPLC-1 are recommended for quality assurance.

#### Abrasion Boot

The abrasion boot specimens are polyurethanes compounded with carbon black. After dissolving the polymer in THF, the carbon black particles are removed by high speed centrifugation and filtration of the solution. Low porosity SEC analysis showed that the specimens contain a low MW component in addition to the polymer, and also resolved minor polyurethane oligomer components as shown in Figure 25. The ratio of the integrated peak area of the low MW component to that of the polymer may be determined by Procedure SEC-1 using a UV280 nm detector.

Number-, weight-, and z-average molecular weights of the polyurethanes were determined by Procedure SEC-2 (Figure 26). Since well-characterized, narrow MW distribution fractions of the polyurethane were unavailable, polystyrene standards were used for MW calibration of the retention times (ASTM Standard Test Method D3536). Hence, calculated polyurethane MW averages are relative to the polystyrene standard and are not to be construed as absolute values. The mean and standard deviation (STD DEV) of the MW averages of the nine abrasion boot specimens are shown in Table 4. The heterogeneity index  $M_w/M_n$  provides an indication of the breadth of the polymer MW distribution. Percent standard deviations and the ranges between the largest and smallest values are relatively small for this type of analysis, indicating that there are no significant differences in the MWs and MW distributions of the specimens.

Table 4. MOLECULAR WEIGHT PARAMETERS ABRASION BOOT POLYURETHANE SPECIMENS

	$M_n$ (g/mol)	$M_w$ (g/mol)	$M_z$ (g/mol)	$M_w/M_n$
Average	59,100	113,000	191,000	1.92
STD DEV	4,500	4,000	15,000	0.10
(% STD DEV)	(7.6)	(3.5)	(7.9)	(5.2)
Largest Value	66,300	118,000	215,000	2.03
Smallest Value	53,000	105,000	171,000	1.74
ESTANE #147	44,800	85,700	136,000	1.91

An additional abrasion boot specimen designated ESTANE #147 was received for analysis at seven months. Reportedly, that particular abrasion boot specimen failed to pass the rain erosion test. Using SEC analysis, it was noted that the MW averages for ESTANE #147 (see Table 4) were appreciably less than the respective values obtained for specimens 5AB1 through 5AB9. It is possible that the failure of the abrasion boot was related to the somewhat lower MW of the uncured ESTANE #147 specimen.

Procedures SEC-1 and SEC-2 are recommended for quality assurance.

## Balance Weight Adhesive

The balance weight adhesive specimens consist of polychloroprene (neoprene) elastomer compounded with carbon black. To prepare samples for HPLC analysis, the polymer is dissolved in THF and carbon black particles are removed by centrifugation and filtration. SEC analysis of solutions showed that the specimens contain more than six low MW components (Figure 27) and that the relative amounts of the low MW components vary widely from specimen to specimen.

Procedure SEC-2 was employed with Universal calibration (ASTM Test Method D3593) for analyzing MW averages. Mark-Houwink constants used in the MW determinations are  $K = 4.18 \times 10^{-3}$  and  $a = 0.83$  for polychloroprene and  $K = 8.61 \times 10^{-3}$  and  $a = 0.74$  for the polystyrene standard. A typical SEC chromatogram for the MW analysis of the polymer is shown in Figure 28 and MW parameters are listed in Table 5. The precision of the SEC procedure was determined from repetitive analysis of the samples and is indicated by the STD DEV and % STD DEV of the MW averages. It is noted that the MW parameters for a number of specimens exceed the error in measurement. However, it is doubtful that the differences are great enough to affect properties of the balance weight adhesive.

Table 5. MOLECULAR WEIGHT PARAMETERS BALANCE WEIGHT ADHESIVE SPECIMENS (NEOPRENE)

Specimen	M <sub>n</sub> (g/mol)	M <sub>w</sub> (g/mol)	M <sub>z</sub> (g/mol)	M <sub>w</sub> /M <sub>n</sub>
5AC1	65,800	144,000	197,000	2.19
5AD2	53,400	117,000	159,000	2.19
5AD3	54,100	136,000	195,000	2.51
5AD4	57,300	128,000	180,000	2.23
5AD5	57,600	127,000	177,000	2.20
5AD6	82,400	189,000	256,000	2.29
5AD7	74,100	182,000	241,000	2.46
Precision				
STD DEV	3,200	2,200	4,800	
(% STD DEV)	(5.3%)	(1.7%)	(2.5%)	

## SUMMARY

HPLC test methods for the quality assurance of organic materials used in the manufacture of certain helicopter rotor blades are recommended in this report. The test procedures are described in the Experimental Section and may be run using commercially available liquid chromatography equipment. Specimens of 12 difference materials were received over a period of three years and analyzed using the recommended methods. Conclusions from the analyses are summarized below.

### Epon 828

There were only very slight differences in the compositions of the Epon 828 specimens. It is unlikely that the differences would have a significant affect on the processability or performance of items manufactured from the resin.

## **BDMA/MTHPA**

The composition of BDMA/MTHPA mixtures is sensitive to storage and handling conditions and will change with time following mixing. In particular, MTHPA is susceptible to hydrolysis. Differences in BDMA/MTHPA composition may be reflected as differences in the processability of resins and the properties of items manufactured from resins containing BDMA/MTHPA as the curing agent. The compositions of BDMA/MTHPA mixtures received as part of this project were appreciably different from one another. It is recommended that HPLC test methods be applied as soon as possible after the BDMA/MTHPA mixtures are prepared and before they are used.

## **Spline Winding Resin**

Differences were apparent in the compositions of various spline winding resin specimens. Differences were in the amounts rather than in the types of components in the resins, and were attributed primarily to problems in sampling. A special sampling procedure is required for this material to avoid crystallization of certain components.

## **Spline Curing Agent**

There were appreciable specimen-to-specimen variations in the concentrations of curing agent components. However, more knowledge of the relationship between the composition of the curing agent and the properties of the cured resin is needed to determine whether the variations are significant.

## **Skin Material**

There was a major change in the chemical composition of the skin material part way through this project. The compositions of the first six specimens were essentially identical to one another, but were quite different from that of the final three specimens. The change in composition was attributed to a change in supplier.

## **STA 66 Doubler**

There was a major change in the chemical composition of the doubler material part way through this project. The compositions of the first six specimens were very similar to one another, but were quite different from that of the final three specimens. The change in composition was attributed to a change in supplier.

## **Adhesive A**

There were no changes in the types of components used in the adhesive formulation; however, differences in the relative amounts of components were evident in some of the specimens. The analyses indicated that the several specimens were somewhat more advanced or partially reacted than others. Research relating chemical composition to processability and properties of the cured adhesive is required to determine the significance of the variations in adhesive composition, and to develop criteria for quality assurance.

**Primer A**

No changes in components were detected and variations in the relative amounts of the primer components were negligible.

**Adhesive B**

No changes in the types of adhesive components were detected; however, variations in the relative amounts of components were observed. The type and extent of variations in the adhesive specimens are similar to those determined for Adhesive A. It is noted that the chemical composition of Adhesive B was quite different from that of Adhesive A.

**Primer B**

No changes in the composition of the primer were noted.

**Abrasion Boot**

There were no significant differences in the MWs and MW distributions of the polyurethane abrasion boot specimens. The MW of an additional specimen which is believed to have failed the rain erosion test, however, was found to be appreciably lower than the MWs of the other specimens.

**Balance Weight Adhesive**

The relative amounts of low ME components found in the polychloroprene adhesive varied widely from specimen to specimen. However, there were no significant differences in the MWs and MW distributions of the polymers.

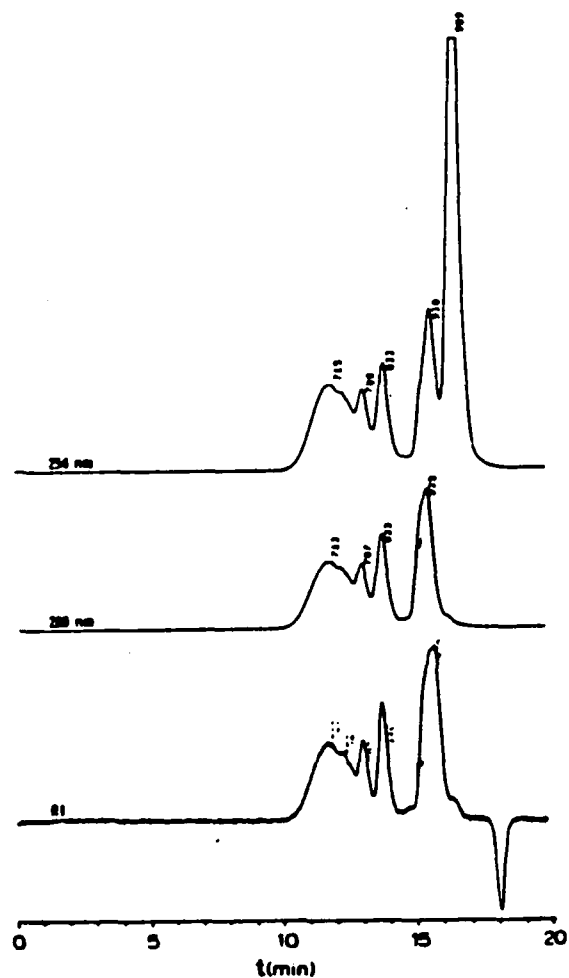


Figure 1. SEC analysis of film Adhesive A. Procedure SEC-1. Columns: micro-Styragel (1000, 500, 2 x 100 Å). Detectors: UV254 nm, UV280 nm, and RI.

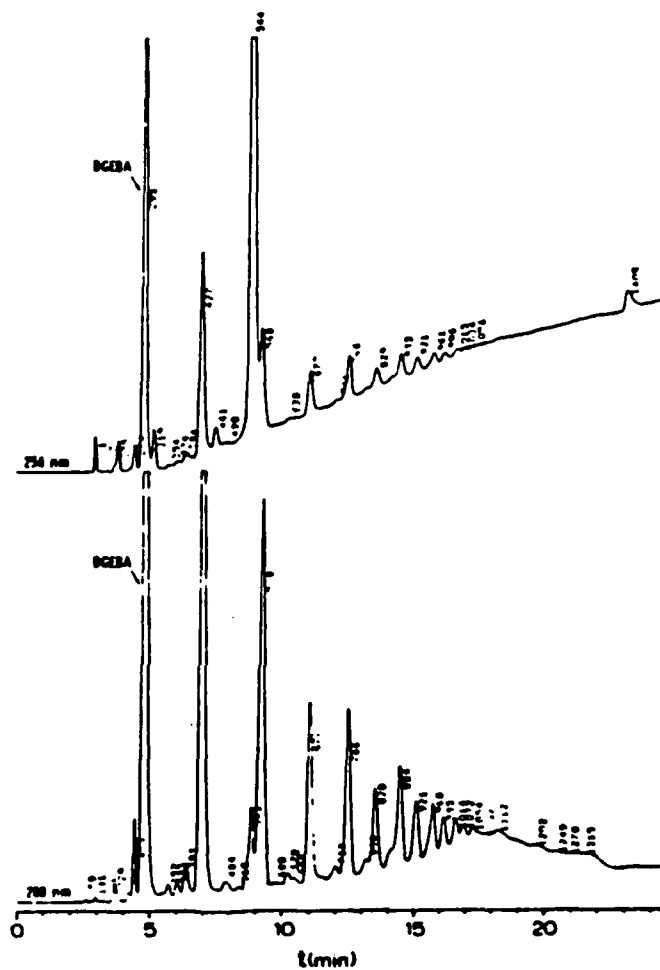


Figure 2. HPLC analysis of film Adhesive A. Procedure HPLC-1. (60% C8/40% THF) to 100% THF, 30 min, gradient 6. Detectors: UV254 nm and UV280 nm.

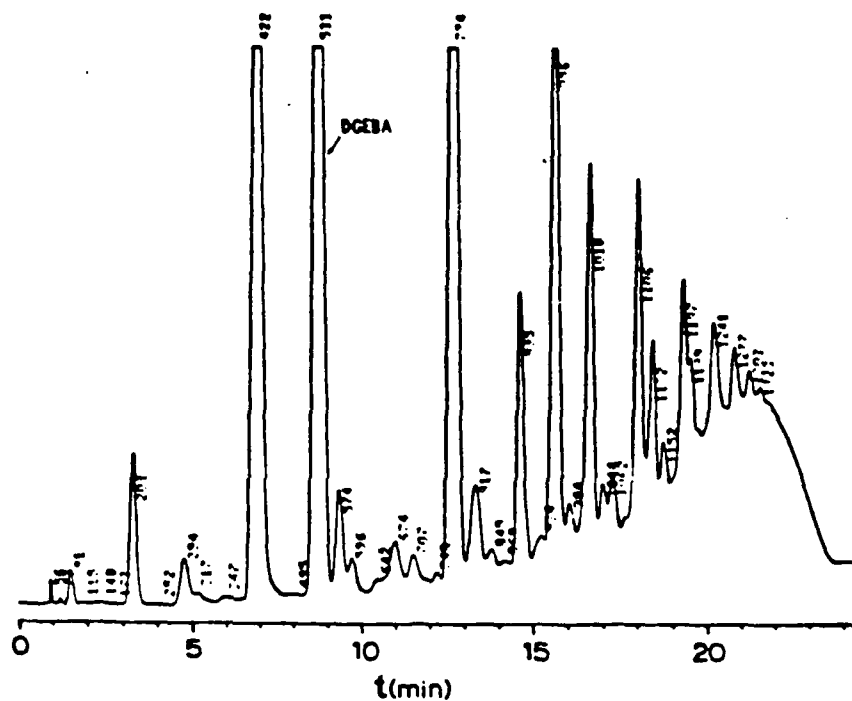


Figure 3. RPHPLC analysis of film Adhesive A. Procedure RPHPLC-1. (60%  $H_2O$ /40% THF) to 100% THF, 30 min, gradient 6. Detector: UV280 nm.

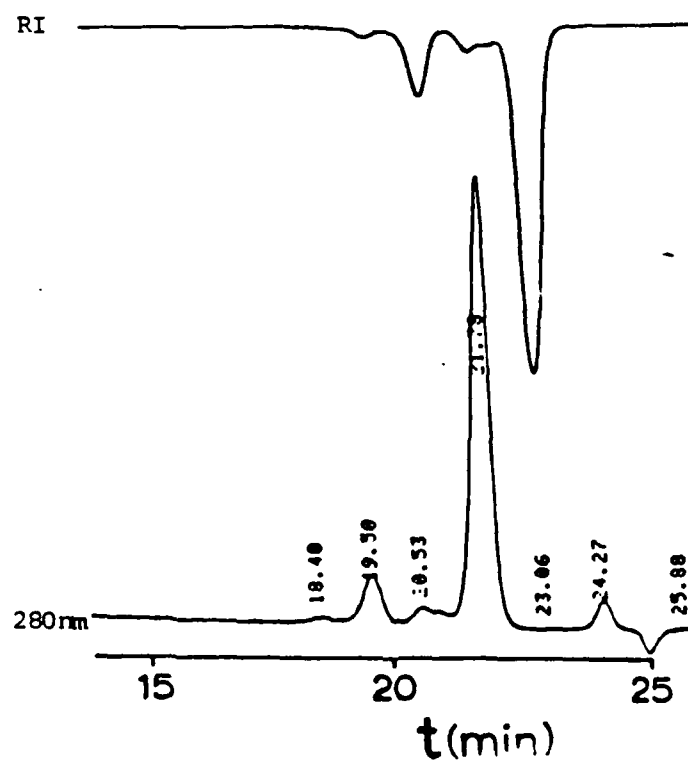


Figure 4. SEC analysis of Epon 828. Procedure SEC-1. Columns: micro-Styrigel (1000, 2 x 500, 2 x 100 Å). Detectors: UV280 nm and RI.

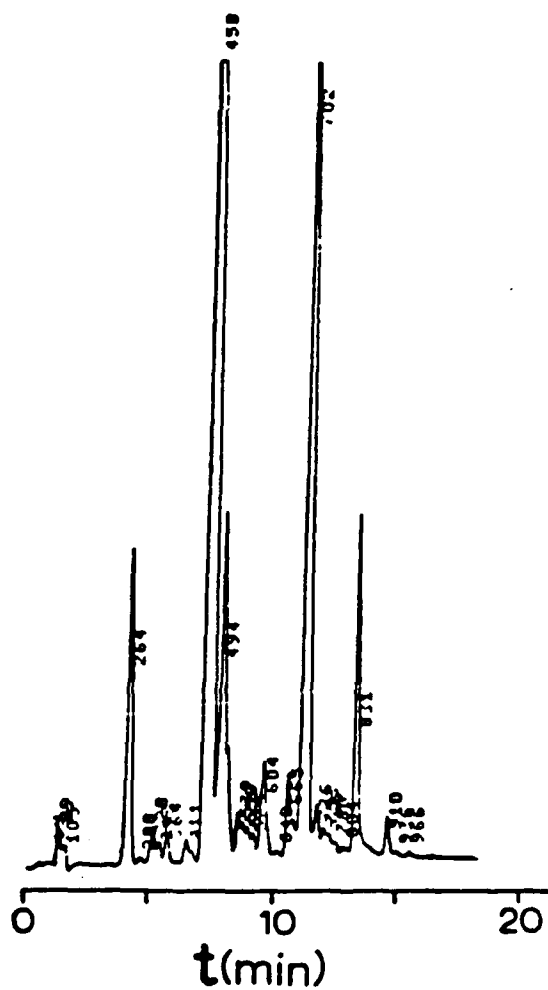


Figure 5. RPHPLC analysis of Epon 828.  
Procedure RPHPLC-1.

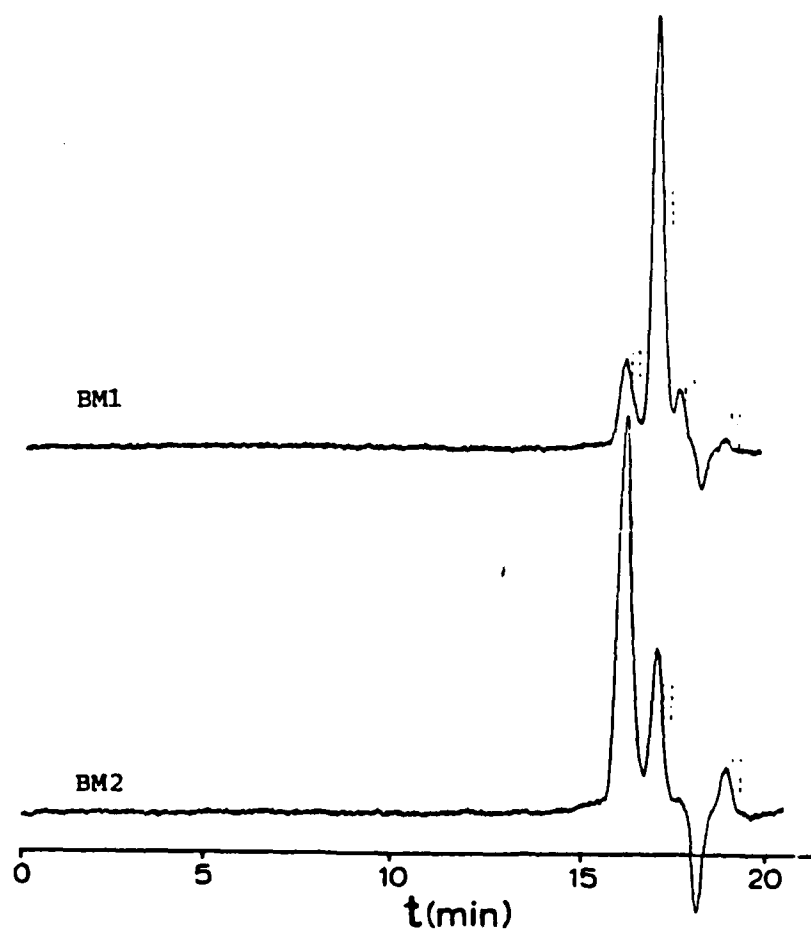


Figure 6. SEC analysis of BDMA/MTHPA specimens BM1 and BM2. Procedure SEC-1. Columns: micro-Styragel (1000, 500, 2 x 100 Å). Detector: RI.

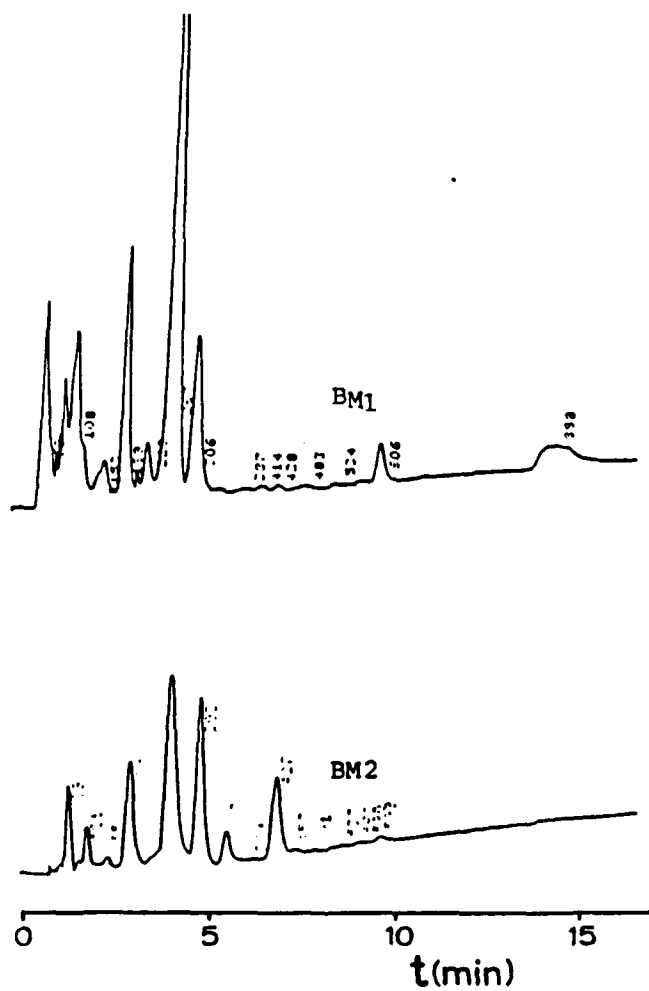


Figure 7. RPHPLC analysis of BDMA/MTHPA specimens BM1 and BM2. Procedure RPHPLC-1. (60% H<sub>2</sub>O/40% THF) to 100% THF, 30 min, gradient 6. Detector: UV280 nm.

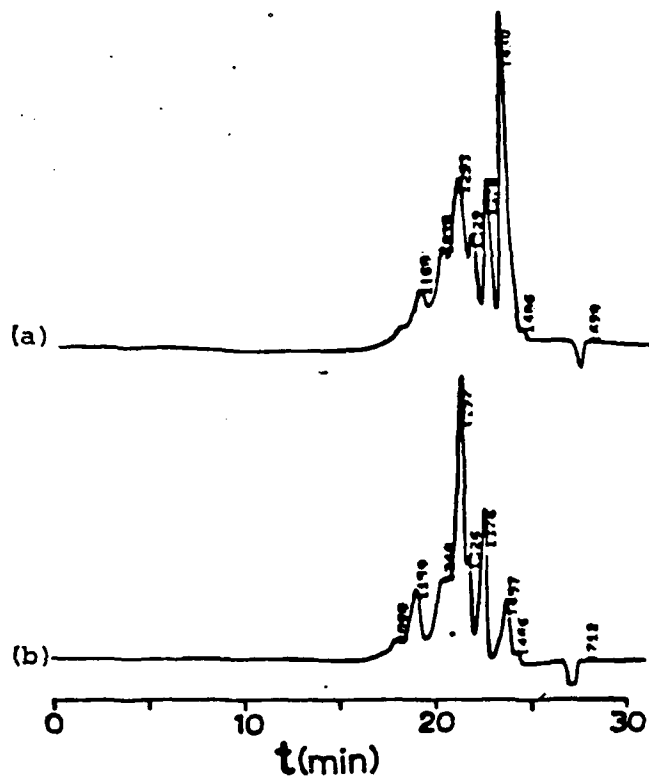


Figure 8. SEC analysis of the spline winding resin (a) sampled as a liquid and (b) sampled as a precipitate. Procedure SEC-1. Column: micro-Styragel (1000, 2 x 500, 2 x 100 Å). Detector: UV254 nm.

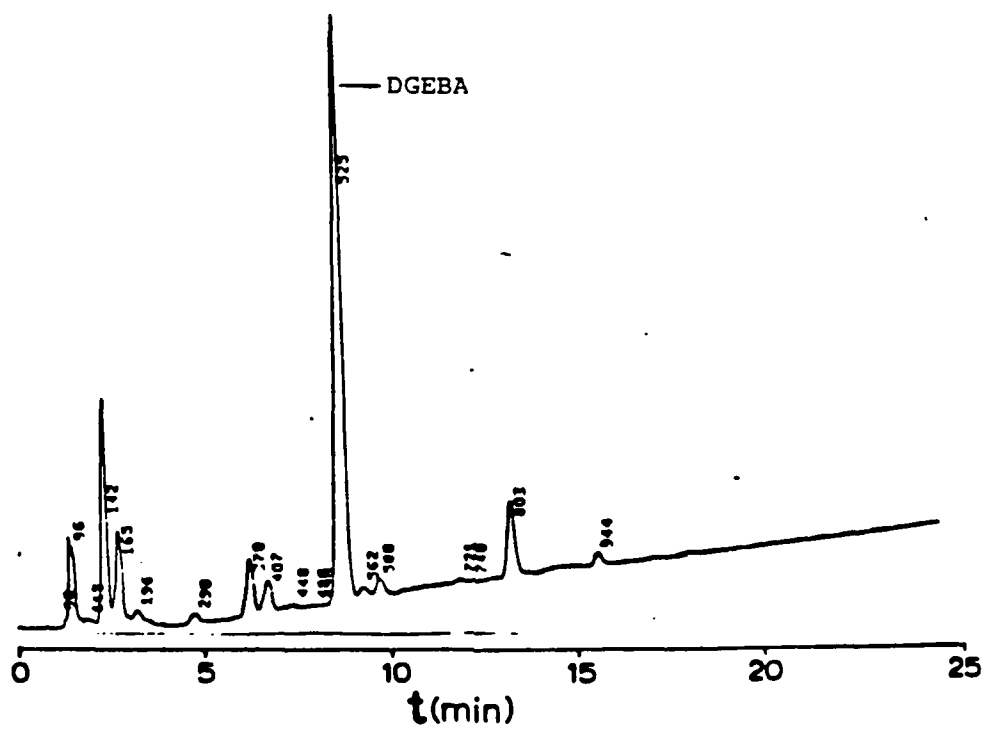


Figure 9. RPHPLC analysis of the spline winding resin. Procedure RPHPLC-1. (60% H<sub>2</sub>O/40% THF) to 100% THF, 30 min, gradient 6. Detector: UV280 nm.

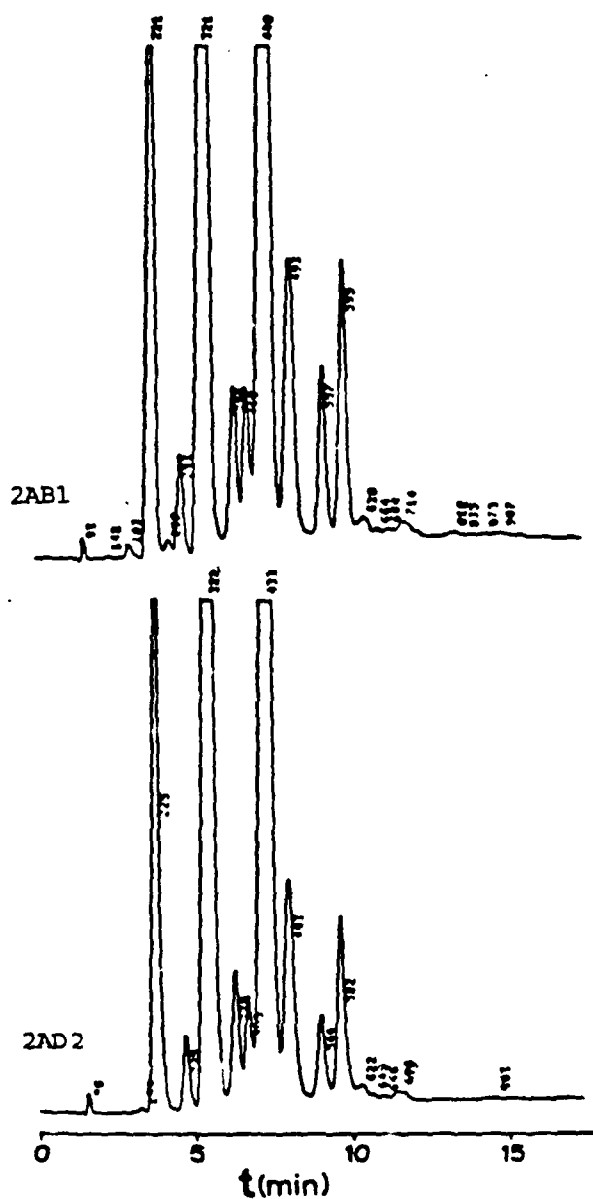


Figure 10. RPHPLC analysis of spline curing agent specimens 2AB1 (0 month) and 2AD2 (7 months). Procedure RPHPLC-1. (60% H<sub>2</sub>O/40% THF) to 100% THF, 30 min, gradient 6. Detector: UV280 nm.

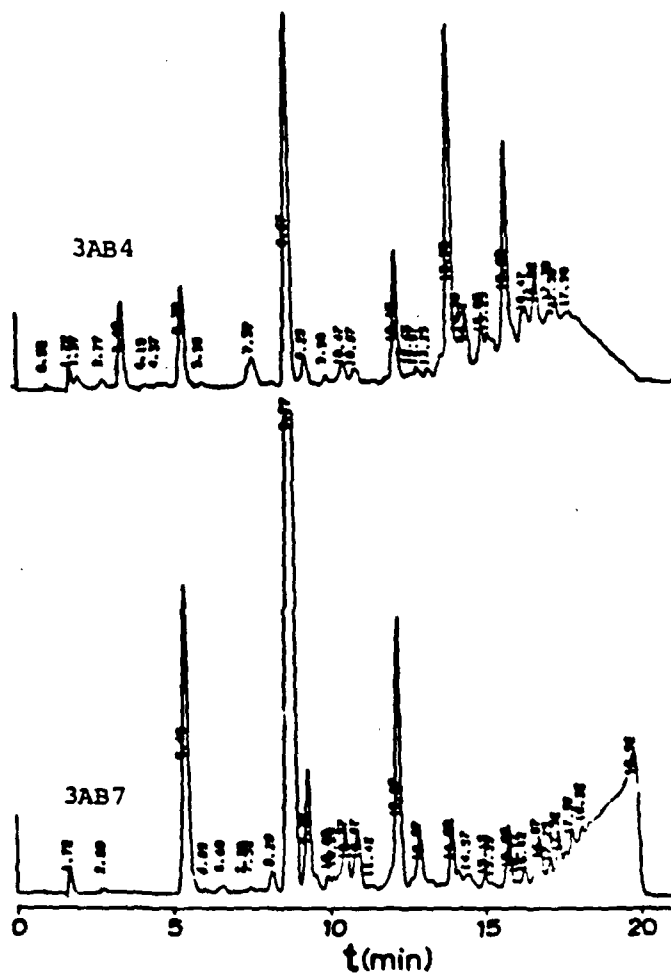


Figure 11. RPHPLC analysis of skin material specimens 3AB4 and 3AB7. Procedure RPHPLC-1. (60% H<sub>2</sub>O/40% THF) to 100% THF, 25 min, gradient 6. Detector: UV280 nm.



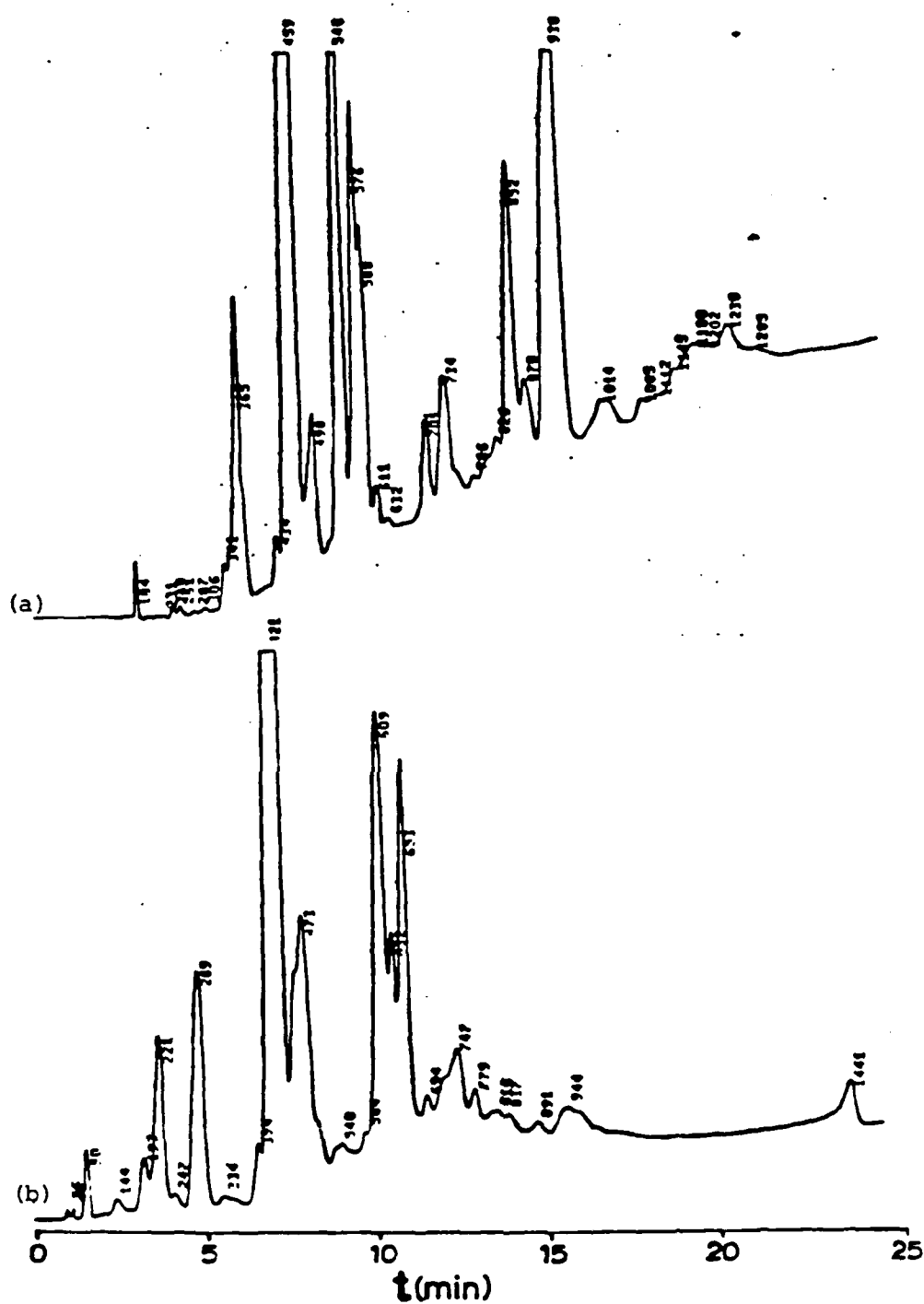


Figure 13. Analysis of STA 66 doubler specimen 4AA1 using (a) procedure HPLC-1, (60% C8/40% THF) to 100% THF, 30 min, gradient 6 with UV254 nm detector and (b) procedure RPHPLC-1 (60% H<sub>2</sub>O/40% THF) to 100% THF, 30 min, gradient 6 with UV280 nm detector.

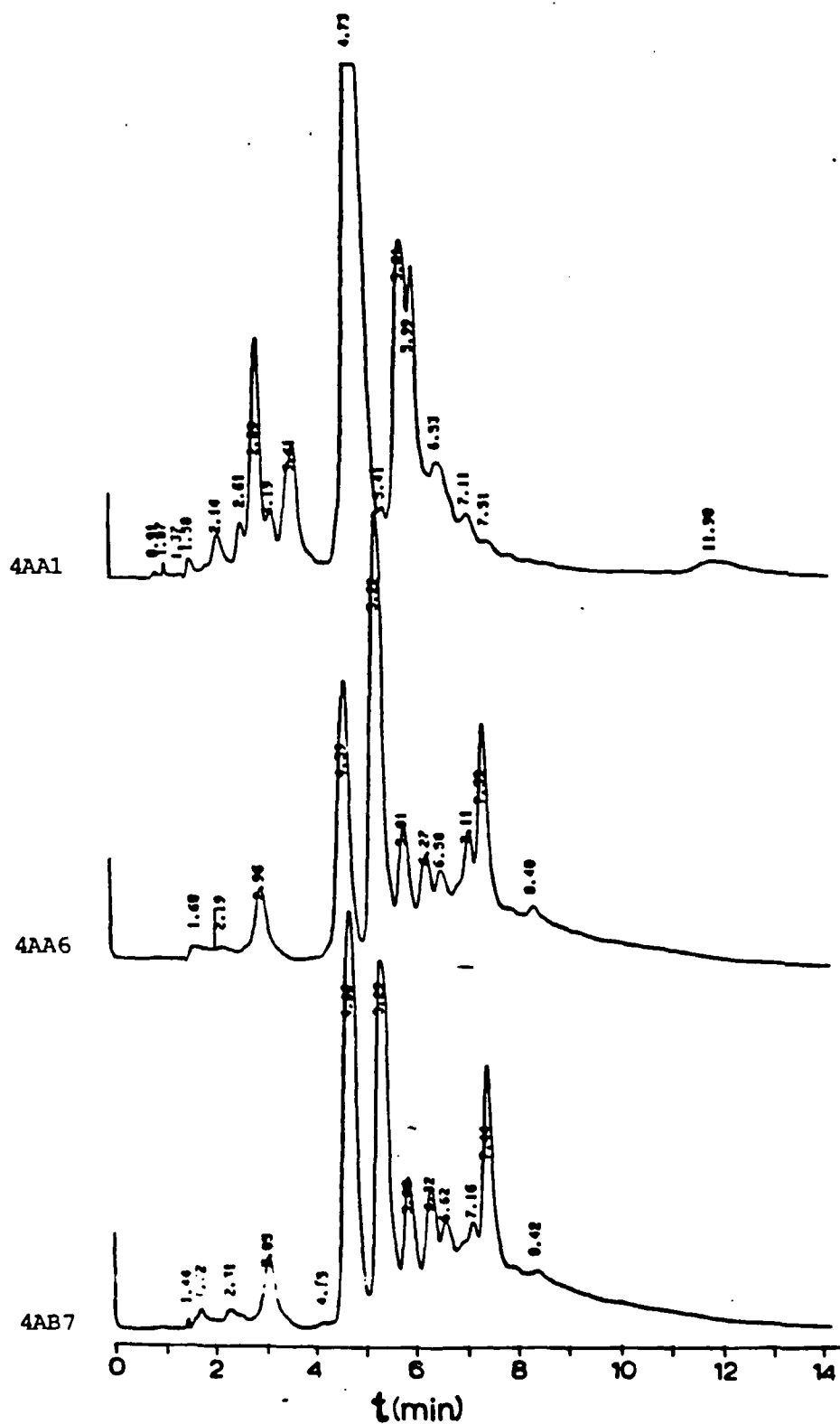


Figure 14. RPHPLC analysis of STA 66 doubler specimens 4AA1, 4AA6, and 4AB7. Procedure RPHPLC-1. (55% H<sub>2</sub>O/45% THF) to (15% H<sub>2</sub>O/85% THF), 30 min, gradient 5. Detector: UV280 nm.

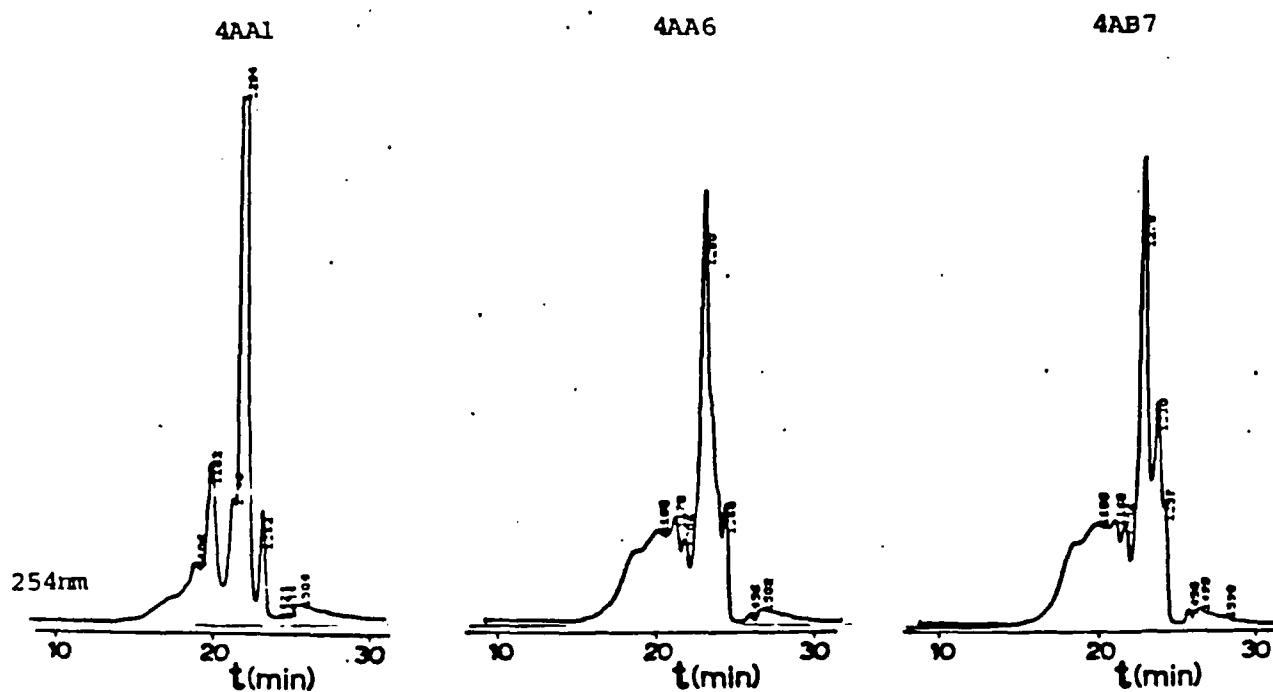


Figure 15. SEC analysis of STA 66 doubler specimens 4AA1, 4AA6, and 4AB7. Procedure SEC-1.  
Columns: micro-Styragel (1000, 2 x 500, 2 x 100 Å). Detector: UV254 nm.

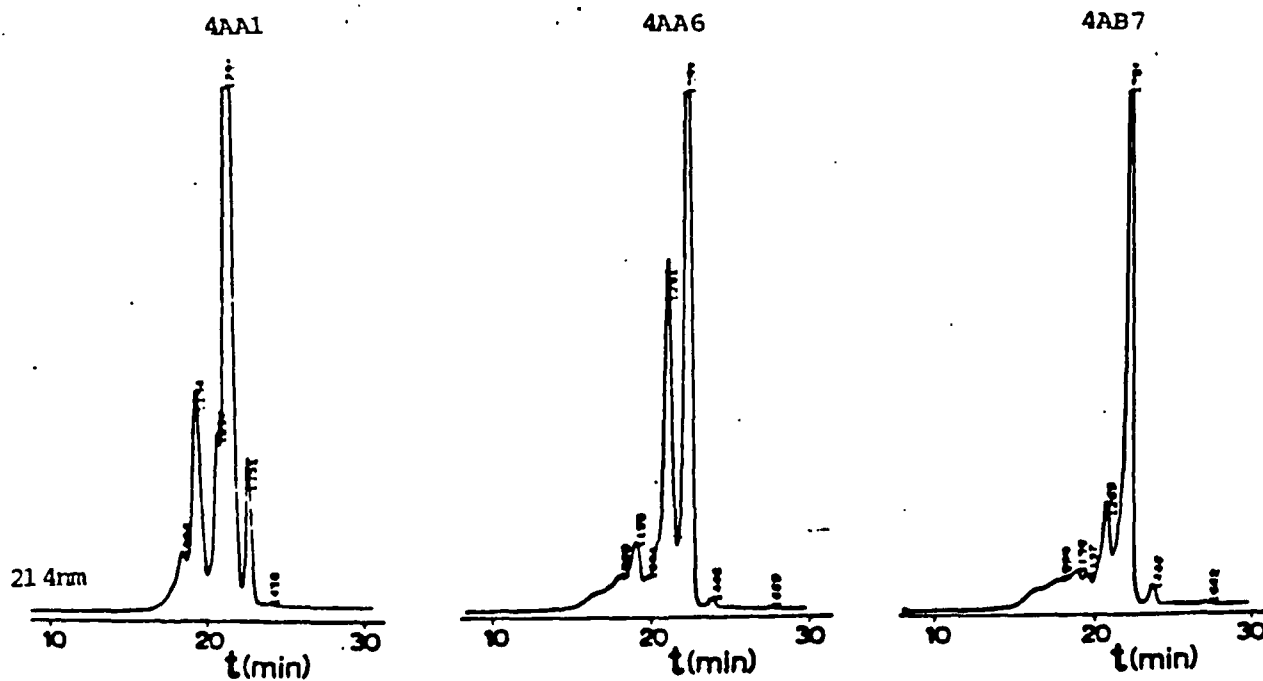


Figure 16. SEC analysis of STA 66 doubler specimens 4AA1, 4AA6, and 4AB7. Procedure SEC-1.  
Columns: micro-Styragel (1000, 2 x 500, 2 x 100 Å). Detector: UV214 nm.

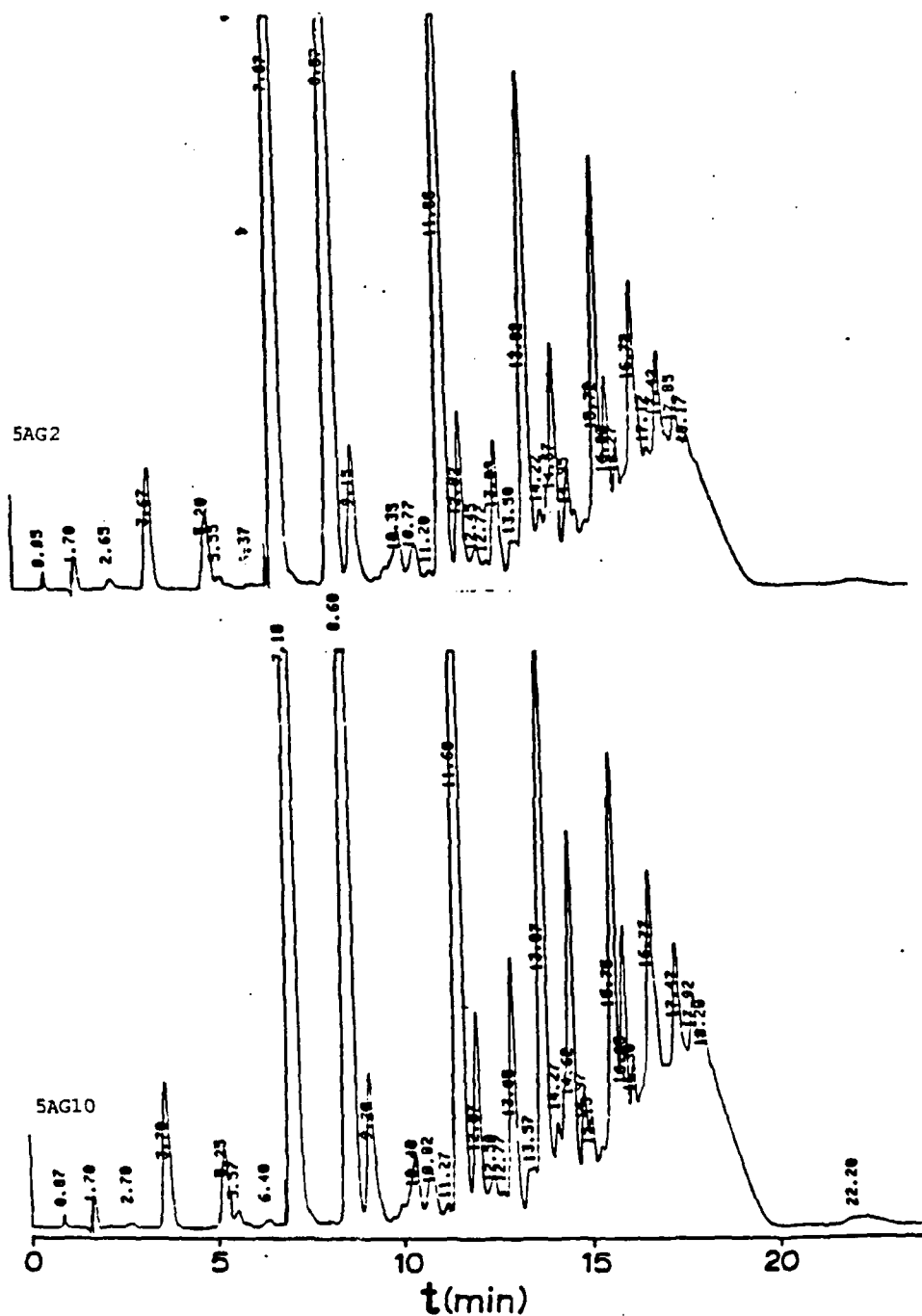


Figure 17. RPHPLC analysis of film Adhesive A specimens 5AG2, 5AG10, 5AG12, and 5AG13. Procedure RPHPLC-1. (60% H<sub>2</sub>O/40% THF) to 100% THF, 25 min, gradient 6. Detector: UV280 nm.

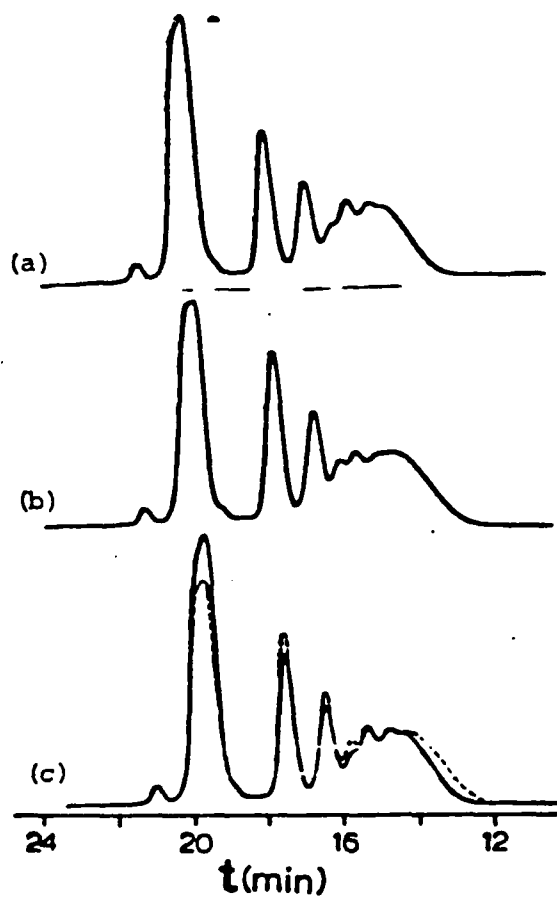


Figure 18. SEC analysis of film Adhesive A specimens (a) 5AG2, (b) 5AG12, and (c) (5AG2) (—) and 5AG12 (----). Procedure SEC-1. Columns: micro-Styragel (1000, 2 x 500, 2 x 100 Å). Detector: UV280 nm.

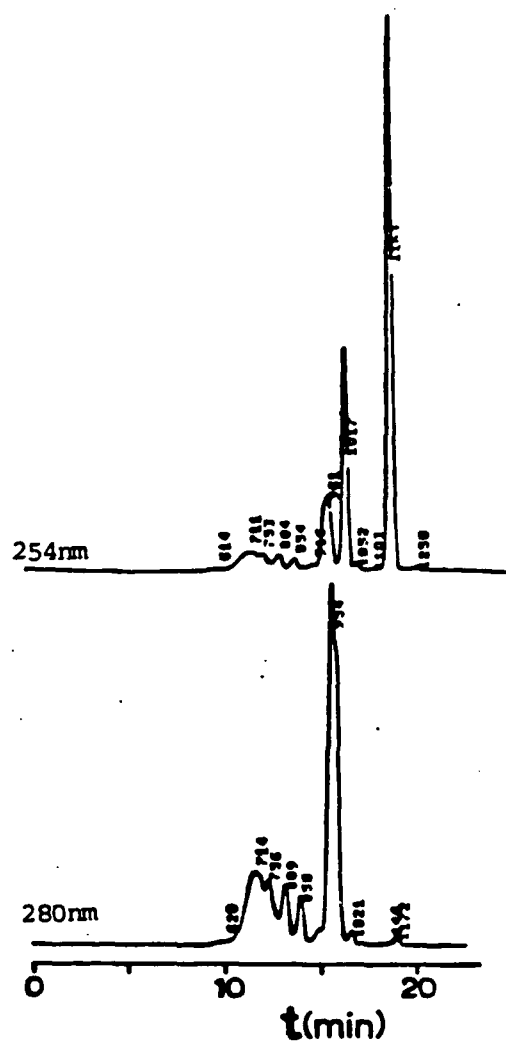


Figure 19. SEC analysis of the Primer A. Procedure SEC-1. Columns: micro-Styragel (1000, 500, 2 x 100 Å). Detectors: UV254 nm and UV280 nm.

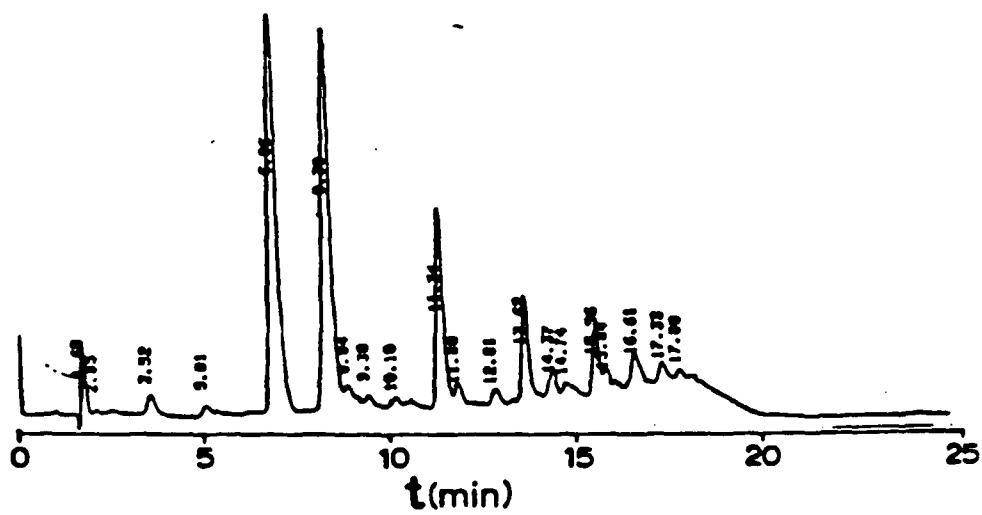


Figure 20. RPHPLC analysis of Primer A. Procedure RPHPLC-1. (60% H<sub>2</sub>O/40% THF) to 100% THF, 25 min, gradient 6. Detector: UV280 nm.

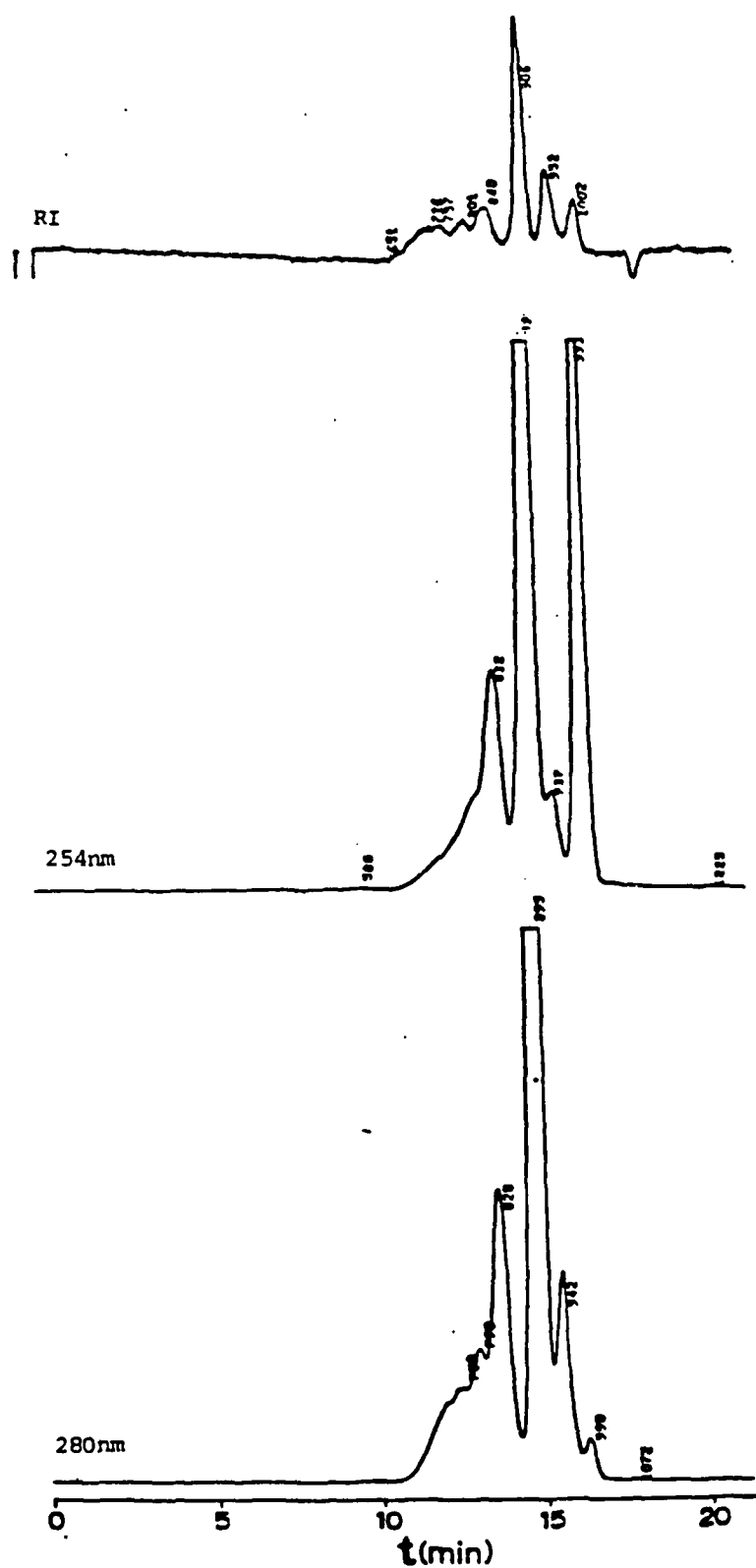


Figure 21. SEC analysis of film Adhesive B. Procedure SEC-1. Columns: micro-Styragel (1000, 500, 2 x 100 Å). Detectors: RI, UV254 nm, and UV280 nm.

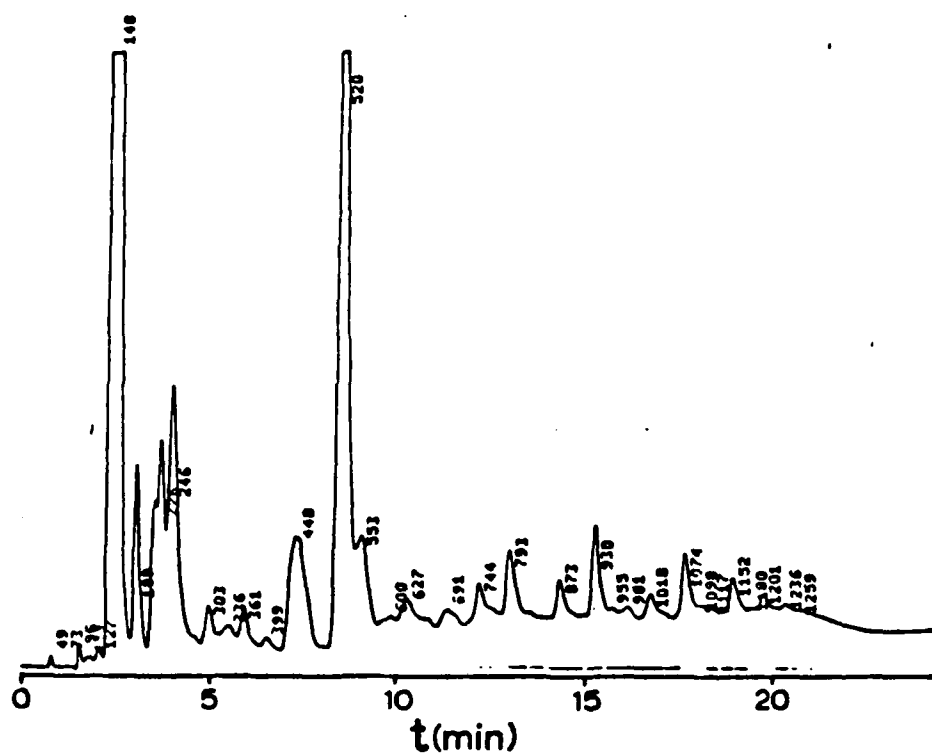


Figure 22. RPHPLC analysis of film Adhesive B. Procedure RPHPLC-1. (60% H<sub>2</sub>O/40% THF) to 100% THF, 30 min, gradient 6. Detector: UV280 nm.

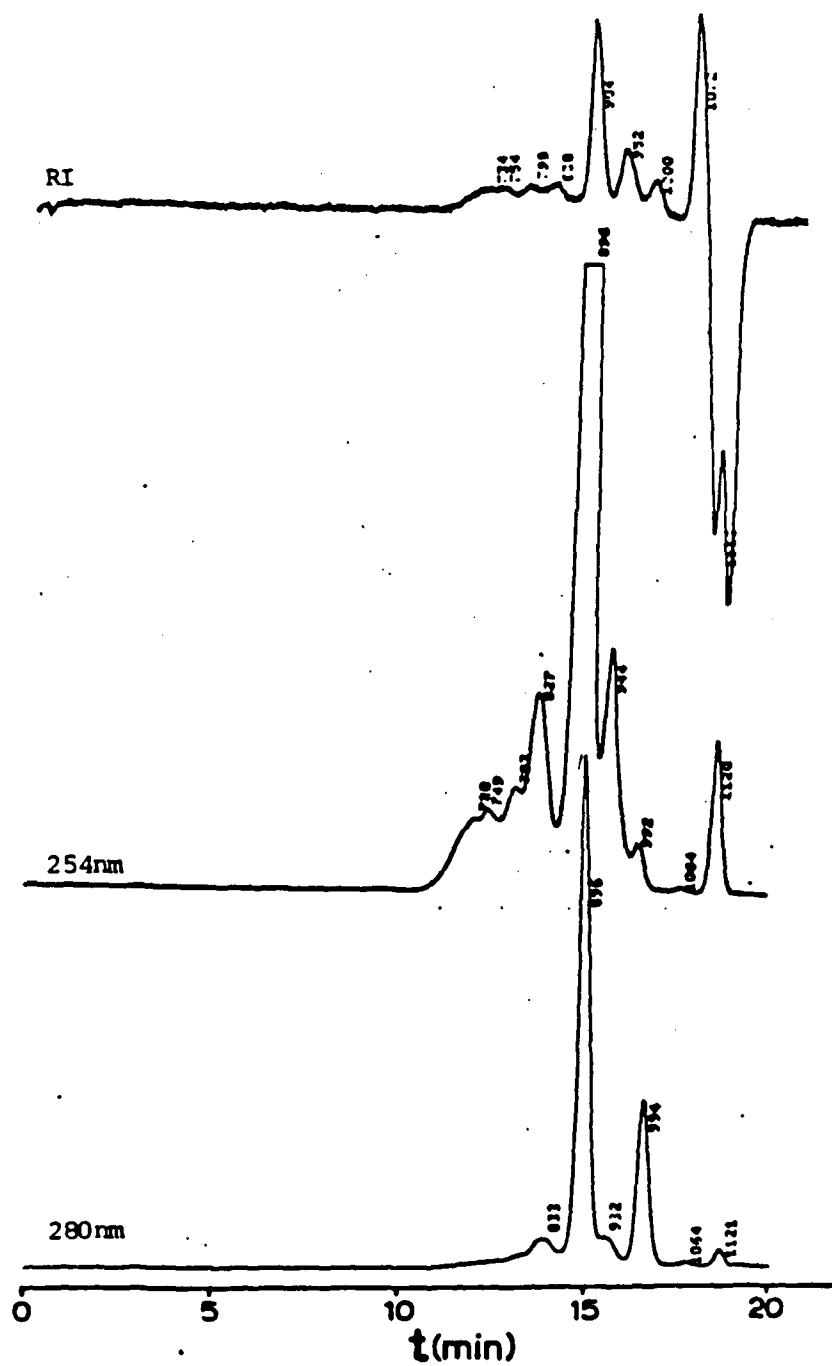


Figure 23. SEC analysis of Primer B. Procedure SEC-1. Columns: micro-Styragel (1000, 500, 2 x 100 Å). Detectors: RI, UV254 nm, and UV280 nm.

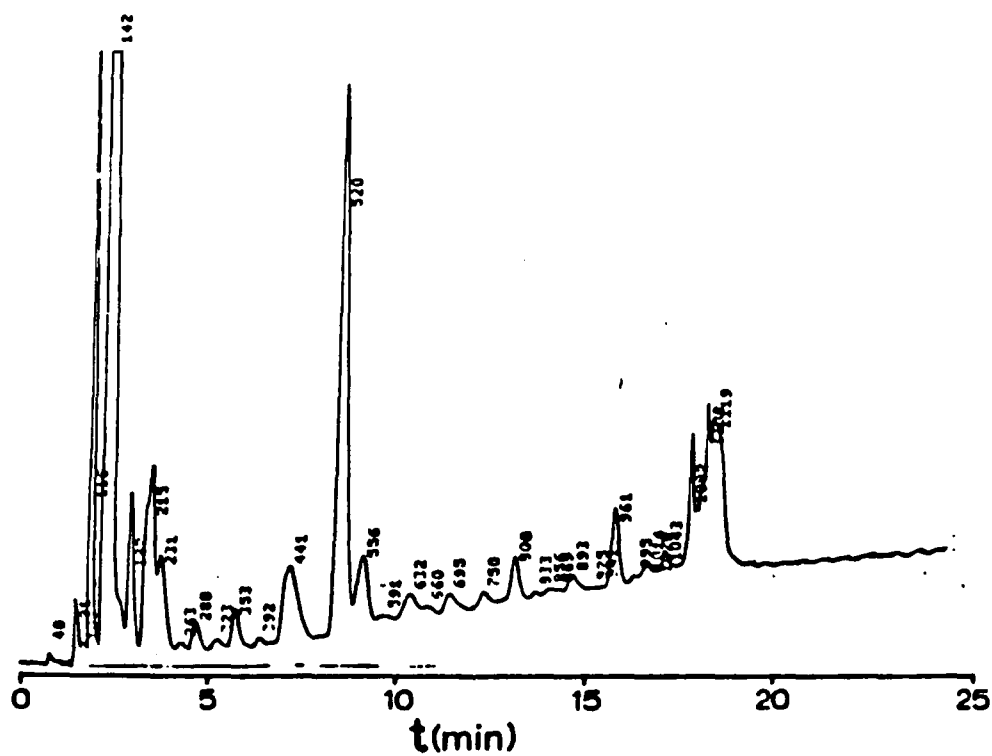


Figure 24. RPHPLC analysis of Primer B. Procedure RPHPLC-1. (60% H<sub>2</sub>O/40% THF) to 100% THF, 30 min, gradient 6. Detector: UV280 nm.

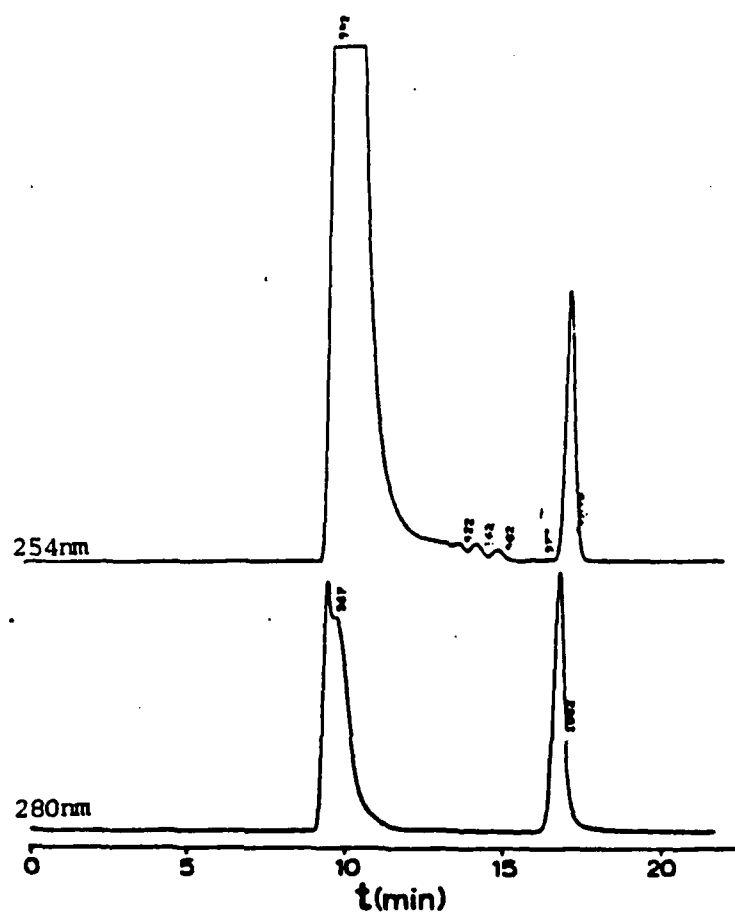


Figure 25. SEC analysis of an abrasion boot specimen. Procedure SEC-1. Columns: micro-Styragel (1000, 500, 2 x 100 Å). Detectors: UV254 nm and UV280 nm.

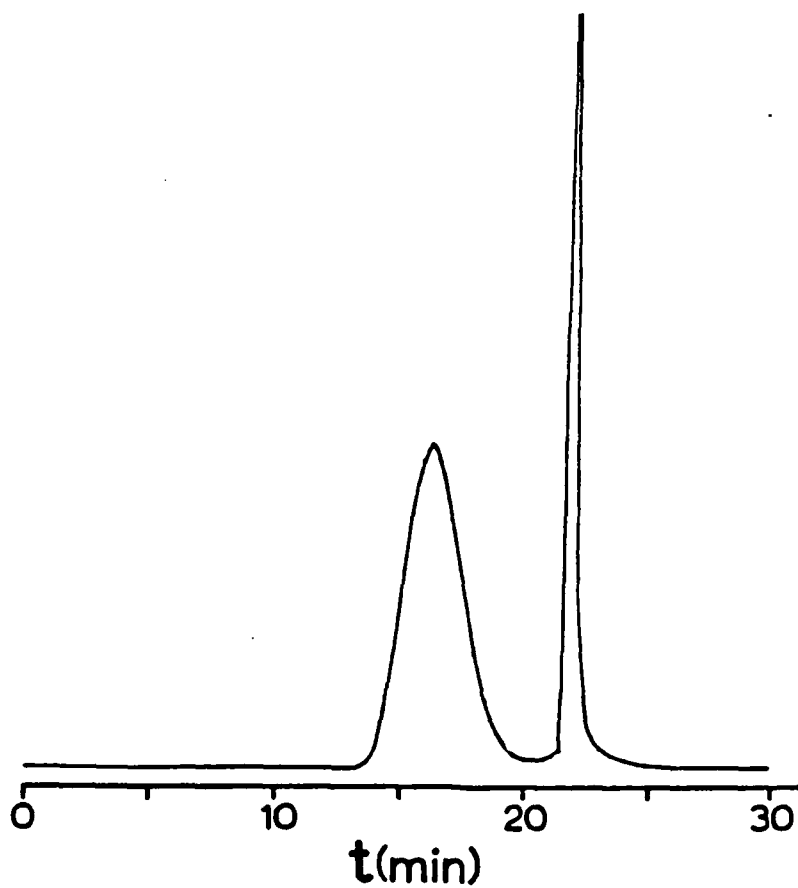


Figure 26. SEC MW analysis of an abrasion boot specimen. Procedure SEC-2. Column: micro-Styragel ( $10^6$ ,  $10^5$ ,  $10^4$ ,  $10^3$  Å). Flow rate: 2 mL/min. Detector: UV280 nm.

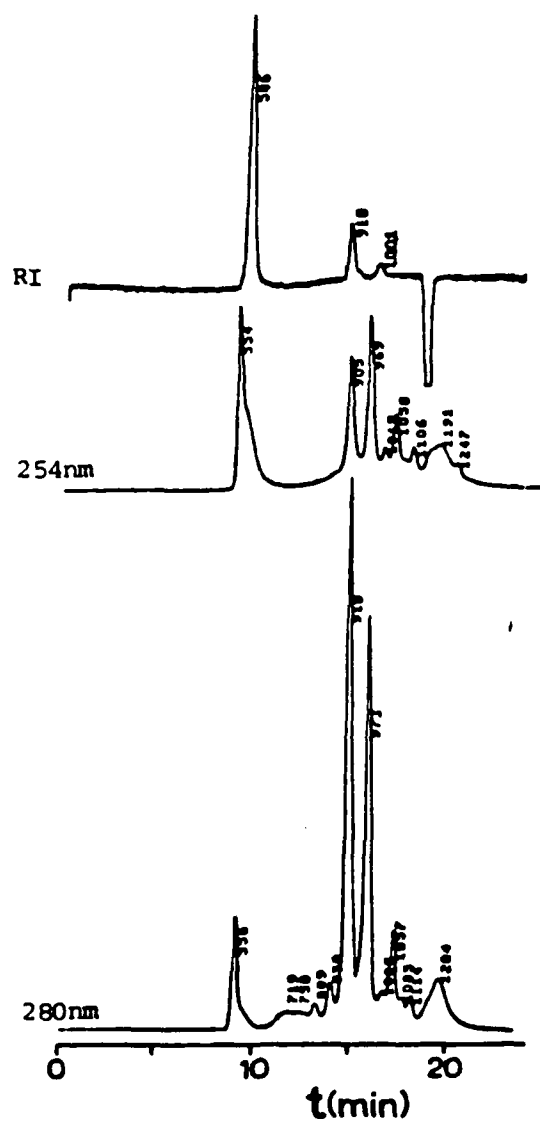


Figure 27. SEC analysis of the balance weight adhesive. Procedure SEC-1. Columns: micro-Styragel (1000, 500, 2 x 100 Å). Detectors: RI, UV254 nm, and UV280 nm.

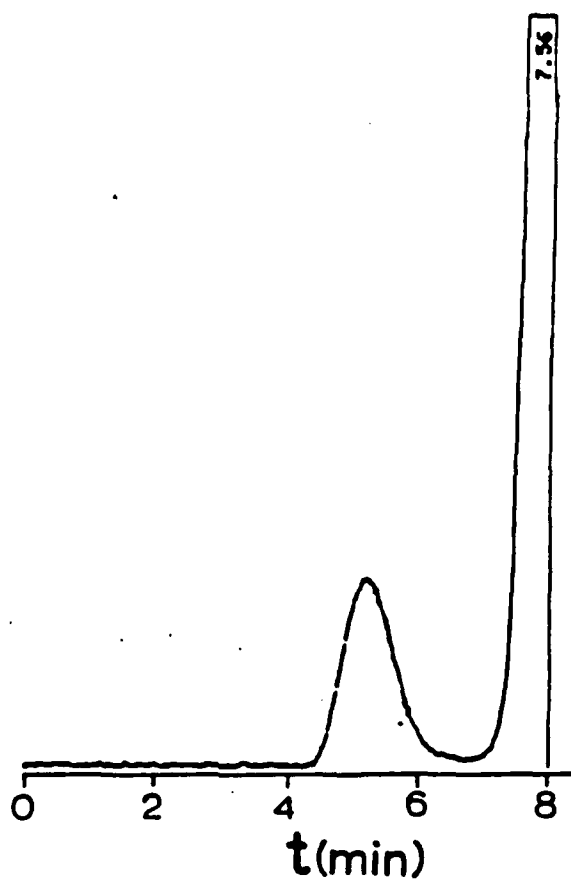


Figure 28. SEC MW analysis of balance weight adhesive. Procedure SEC-2. Columns: Zorbax PSM-1000-S and PSM-60-S. Flow rate: 1.5 mL/min. Detector: RI.

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2	Authors

<p>U.S. Army Materials Technology Laboratory Watertown, Massachusetts 02172-0001 HPLC ANALYSIS OF HELICOPTER ROTOR BLADE MATERIALS - Gary L. Hagnauer and David A. Dunn</p> <p>Technical Report MTL TR 90-12, March 1990, 37 pp- illus-tables, D/A Project: 1L162105.AH84</p> <p>This report describes high performance liquid chromatography (HPLC) test methods developed for the quality assurance of organic materials used in the manufacture of Army helicopter rotor blades. Since the chemical compositions of most of the organic materials were unknown at the start of this project, HPLC was employed to help identify components in specimens. Test methods were developed and then applied to monitor the chemical compositions of specimens received from the manufacturer during blade fabrication. Six HPLC test methods were used to evaluate eight sets of specimens of 12 different materials received over a span of three years. The HPLC test methods included size-exclusion, normal-phase, and reverse bonded-phase chromatography techniques and employed a variety of columns, mobile phases, and detectors. Major changes in chemical composition and variations in relative concentrations of chemical components were noted in seven of the 12 materials monitored during the course of this study. Test results are summarized and recommendations are made.</p>	<p>AD <u>UNCLASSIFIED</u> UNLIMITED DISTRIBUTION</p> <p>Key Words</p> <p>Liquid chromatograph Helicopter blades Testing</p>
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